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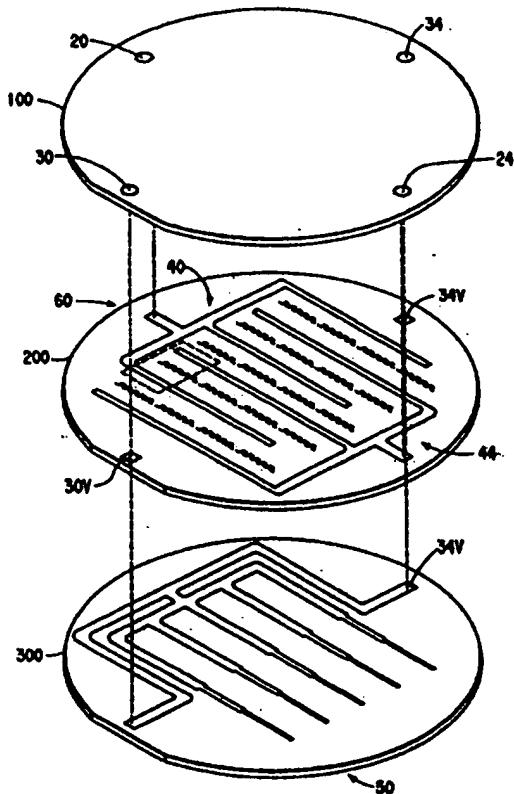
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(54) Title: CHEMICAL MIXING AND REACTION APPARATUS AND PROCESSES FOR THE PREPARATION THEREOF

(57) Abstract

An integral structure is provided for chemical processing, particularly high-speed mixing and chemical reacting, in which a plurality of laminae (100, 200, 300) are joined together and having inlet (20, 24) and outlet ports (30, 34) connected by a plurality of intersecting channels formed therethrough which accommodate the passage of one or more chemicals, said laminae (100, 200, 300) comprising a material selected for compatibility with the one or more chemicals, the channels being formed so that chemicals are combined at intersections therein at sufficient angles of attack and shear rates so that chemical reactions are not limited by mass transfer. Chemicals are introduced through the inlet ports (20, 24) and processed along the channel, with desirable product withdrawn through the outlet ports (30, 34). The laminae are selected from material of groups III, IV or V of the Periodic Table. Processes of manufacture of the apparatus and processes utilizing the apparatus are also disclosed herein.



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TITLE OF THE INVENTIONCHEMICAL MIXING AND REACTION APPARATUS
AND PROCESSES FOR THE PREPARATION THEREOFFIELD OF THE INVENTION

5 This invention relates to an integrated chemical processing apparatus, adapted for use with high-speed chemical reactions, which can be incorporated into a larger integral structure of multiple chemical processing units or an integrated system. More
10 particularly, the present invention is directed to chemical processing apparatus characterized by improved rapid mixing of chemicals passing therethrough, by enhanced safety of operation, and by reduced capital investment.

15 BACKGROUND OF THE INVENTION

To achieve efficient chemical processing, it is necessary to precisely control a number of processing parameters, such as temperature, pressure, mixing conditions, exposure of reactants to catalyst material, 20 exposure of reactants to products and/or byproducts and exposure of reactants to actinic radiation. Certain chemical reactions are particularly difficult to perform in an optimal manner because the chemical reactions occur very quickly, sometimes before the reactants are 25 completely mixed together. Certain non-stoichiometric portions of the partially mixed reactants may cause reaction products other than those desired to be produced.

Conventional chemical processing equipment 30 typically holds a relatively large volume of materials and consequently has a relatively large volume to surface area ratio, and are thus particularly ill-suited for high speed chemical reactions. Different portions of the reactant materials contained within such 35 processing equipment are more likely to be exposed to

different histories of conditions. In the case of a conventional tank reactor, for example, when reactants are introduced they are typically added in separate streams, usually at controlled rates, and are then mixed together. The so-called T-mixer has been used to mix incoming streams together before they enter a reactor tank. For chemical reactions that occur rapidly, i.e., typically in less than one second, insufficient mixing may have occurred, even with use of the T-mixer, before the reaction is well established. Portions of the incompletely mixed mixture may be starved of one or the other reactant and undesired secondary reactions may occur which produce undesired byproducts.

Rapid stirring of the reactants may reduce this mixing history difference, but will not eliminate it. As a result of the nonhomogeneous mixing history, different portions of the reactants may chemically react differently. Undesired reactions may occur in portions of the reactants that cause localized heating in these different portions. This localized heating may accelerate undesired reactions. This may result in the production of undesired waste products, which may be hazardous and/or which must be properly disposed of. In extreme situations, reaction rates may accelerate to uncontrollable levels, which may cause safety hazards, such as potential explosions.

If, however, the volume in each reactant stream being mixed is substantially reduced, then the speed of mixing of the reactants may be greatly increased to substantially improve the control of homogeneity of mixing history of the reactants.

It has been recognized that a high degree of flow turbulence enhances the ability to rapidly mix two or more reactants together. Rapid mixing is known to be important for rapid chemical reactions. A high degree

of turbulence is also known to enhance heat transfer as well as mixing rates. Thus a structure having both a low contained volume and a high degree of flow turbulence is particularly advantageous for precise 5 control of high-speed chemical reactions.

Mixer assemblies having highly turbulent flow have been constructed by machining the desired passages and chambers in metal plates, using conventional metalworking techniques, and then assembling the plates 10 into a stack and either clamping the stack together or permanently joining the stack, as by welding or soldering. An example is U.S. Patent 3,701,619. Structures formed using conventional machine tool techniques cannot economically achieve volume to surface 15 area ratios that are very low. The materials of construction of conventional chemical processing apparatus, such as steel and specialty iron alloys, furthermore may be subject to corrosion and wear, may have undesirable effects on catalytic activity, or may 20 "poison" a catalyst.

It is an object of the present invention to provide a chemical processing unit that mixes reactants in a rapid and efficient manner, such that chemical reactions are not limited by mass transfer considerations. The 25 present invention provides the capability to integrate one or more mixing/reaction units with control elements into a larger integrated chemical processing system to meet the needs of a specific high-speed chemical reaction. A feature of the present invention is that it 30 can be economically used in the laboratory, to make a range of precise sizes of mixing/reaction units, to perform the basic chemical reactions for determining the optimum operating parameters. Commercial production volumes may then be readily achieved by replicating the

mixing/reaction units and operating them in parallel in a larger integrated chemical processing system.

Advantages of the present invention, when used in a larger integrated chemical processing apparatus, include 5 the elimination of many interconnections and joints, thereby reducing the potential for leaks. These and other objects, features and advantages will become better understood upon having reference to the following description of the invention.

10 SUMMARY OF THE INVENTION

There is disclosed and claimed herein an apparatus for use in an integral structure for chemical mixing and reacting of one or more chemicals, comprising a plurality of laminae joined together and having a 15 plurality of intersecting channels formed therethrough which accommodate the passage of one or more chemicals. The laminae comprise a material selected for compatibility with the one or more chemicals, the channels being formed so that chemicals are combined at 20 intersections therein at sufficient angles of attack and shear rates so that chemical reactions are not limited by mass transfer considerations. Moreover, the shear rate is sufficient to disrupt boundary layer formation. The channels are arranged such that two or more channels 25 are positioned at a point of intersection to form an angle of attack of from 20 to 160 degrees, preferably from 70 to 110 degrees, and most preferably about 90 degrees.

The apparatus of the invention may be prepared 30 according to the process of the invention. The process comprises first, processing a plurality of laminae each having a top portion and a bottom portion and a desired thickness, sufficient to form desired pathways thereon or therethrough. The laminae are then stacked and

bonded together in precise alignment to develop channels that intersect at a desired angle of attack.

In the process for preparing the integral structure, the pathways on facing surfaces of adjacent

- 5 laminae generally form passages through the structure in the plane of the laminae having the desired cross-sectional areas. These planar passages are connected with each other and with passages orthogonal to the plane of the laminae which pass through one or more
- 10 laminae to form passages having the desired overall three-dimensional shapes.

The apparatus of the present invention may be used in a method for mixing and reacting of chemicals. The method comprises: (a) introducing one or more chemicals

- 15 into channels of the above-described structure;
- (b) directing the chemicals to traverse the channels;
- (c) coordinating traversal at a traversal rate so that angles of attack and shear rates are sufficient to insure that chemical reactions are not limited by mass
- 20 transfer considerations; and (d) withdrawing reacted product from the channels of the structure.

The invention can be more fully understood from the following detailed description thereof in connection with accompanying drawings.

25 BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a perspective view of an apparatus of the present invention.

Figure 2 is an exploded perspective view of the apparatus, as seen from above, showing the top surfaces 30 of three laminae which are used to form the structure.

Figure 3 is an exploded perspective view of the apparatus, as seen from below, showing the bottom surfaces of three laminae which are used to form the structure.

Figure 4 is an enlarged first sectional view, taken along section lines 4-4 of Figure 1.

Figure 5 is an enlarged second sectional view, taken along section lines 5-5 of Figure 1.

5 Figure 6 is an exploded perspective view of a portion of the apparatus of Figure 1, showing the bottom surface of a first lamina 100 and the top surface of the second lamina 200. Figures 6, 8, 9 show a first embodiment arrangement of channels that form an array of 10 mixing elements and a distribution manifold.

Figure 7 is an exploded perspective view of the bottom of a second lamina 200 and the top of a third lamina 300 showing channels that form a distribution manifold.

15 Figure 8 is an enlarged view of the portion of Figure 6 designated as 8-8 showing mixing element 64A2.

Figure 9 is an enlarged view of a portion of Figure 8 designated as 9-9.

20 Figure 10, which corresponds in scale to Figure 9, shows a second embodiment arrangement of channels that form an array of mixing elements and a distribution manifold.

25 Figure 11 is a simplified perspective view which illustrates the channels of Figure 8 and the intersections of those channels.

Figure 12 is a simplified perspective view which illustrates the channels of Figure 10 and the intersections of those channels.

30 Figure 13a-c is a plan view of the channels of Figure 9 showing the channels separately and superimposed.

DETAILED DESCRIPTION OF THE INVENTION

The present invention is characterized by small channels of complex three-dimensional shapes which:

35 (1) can create a high degree of flow turbulence that

enhances rapid mixing and heat transfer; (2) intersect with other channels in a shearing fashion at a predetermined angle of attack; (3) have a very low volume to surface area ratio that minimizes temperature gradients and further enhances heat transfer; and (4) controls residence time of materials therein, to achieve more precise temperature control, and a more uniform temperature history, for every portion of the entire volume of reactants mixed and reacted. Channels small enough that they will not allow the propagation of a flame may be readily formed and thus can be used to safely react potentially explosive chemical reactants. The channels may measure as small as about 10 micrometers in cross section.

Throughout the following detailed description, reference characters between 1 and 99 refer to overall features of the invention. That is, these features may only be apparent on joinder of adjacent laminae. For example, in Figure 7 chamber 50M is comprised of chamber 250M on laminae 200 and chamber 350M on laminae 300. Laminae are numbered 100, 200, 300, and specific features on each respective lamina are numbered 101 to 199, 201 to 299, etc. with the last two digits corresponding to the overall feature of the invention. The suffix letter "V" is used to designate interlamina vertical pathways, also known as "vias", through the structure. Similarly, suffix "M" indicates a manifold chamber within a manifold; suffixes "S" and "T" indicate straight and turning portions of pathways. Suffixes comprising a hyphen and numeral (-1, -2, etc.) are used to designate parts of specific elements, such as individual branches of the branched manifolds. A capital letter and number are appended to the reference character to designate elements of an array (e.g., mixer 64A2 of array 64). Numerals enclosed in curved braces

() designate crystal planes in a crystalline material (e.g., {100}).

Referring to Figure 1, there is shown an apparatus 10 exemplary of the present invention. This apparatus 5 10 is comprised of a plurality of laminae 100, 200, 300 fused together to form an integral structure. One or more inlet ports (here, inlet ports 20 and 24 are shown) enable the flow of reactants into the apparatus and one or more outlet ports (here, outlet ports 30 and 34 are 10 shown) enable the flow of the resulting reaction products out of the apparatus. It is to be appreciated that the inlet ports 20 and 24 and outlet ports 30 and 34 do not necessarily have to be positioned only through lamina 100 as shown but may extend through all three 15 laminae (e.g., outlet port 34 in Figure 4). These elements could be arranged to meet the integral structure at the side of a lamina, for example (not shown). The laminae may be comprised of either the same or different materials. The laminae (also known as 20 wafers) 100, 200, 300 preferably are comprised of materials from groups III, IV or V of the Periodic Table, silicon being the most preferred material. If desired one or more of the laminae may be comprised of an alternate material, such as ceramic, a glass material 25 such as Pyrex or other compatible materials.

Figure 2 shows the top surfaces of the three laminae 100, 200, 300 and Figure 3 shows the bottom surfaces of the three laminae 100, 200, 300 which are used to form the apparatus of Figure 1. Operative 30 features of the apparatus shown in Figures 2 and 3 are: two inlet distribution manifolds 40, 44 formed by pathways in the bottom surface of lamina 100 and the top surface of lamina 200; an array 60 of mixer/reaction chambers represented by a series/parallel arrangement of 35 T-mixers 62 and intersecting-channel mixing elements 64

formed by corresponding pathways in the bottom surface of lamina 100 and the top surface of lamina 200; a fork-shaped outlet collection manifold 50 formed by corresponding pathways in the bottom surface of lamina 5 200 and the top surface of lamina 300.

In sectional views of Figures 4 and 5 the vertical scale is exaggerated for clarity of illustration. Although the laminae of the apparatus are fused into an integral structure when completely fabricated, for 10 clarity of illustration the interfaces between the laminae are shown in Figures 4 and 5.

In Figures 4 and 5, which illustrate typical flow passages in the interior of the structure, the vertical pathways 20V, 24V, 30V, 34V which connect respectively 15 with the inlet ports 20, 24 and outlet port 30 in lamina 100 and outlet port 34 in outer laminae 100 and 300, are typically formed by grinding or drilling through the lamina.

In Figure 4 three passages 40-1, 40-2, 40-3, which 20 are branches of distribution manifold 40 and three passages 44-1, 44-2, 44-3, which are branches of distribution manifold 44 are formed in the top surface of lamina 200. Five passages 50-1, 50-2, 50-3, 50-4, 50-5, which are branches of collection manifold 50, are 25 respectively formed by corresponding mirror image pathways 250-1, 250-2, 250-3, 250-4, 250-5 (see also Figure 7) in the bottom surface of lamina 200 and pathways 350-1, 350-2, 350-3, 350-4, 350-5 (Figure 7) in the top surface of lamina 300.

30 In Figure 5 the central axis of a third horizontal passage 50-3 (see also Figure 8), which comprises the center branch of a 5-branch collection manifold 50, lies in the plane of the sectional view. The horizontal passage 50-3 is formed by corresponding mirror image 35 pathways, respectively pathway 250-3 (Figure 7) in the

bottom surface of lamina 200 and pathway 350-3 (Figure 7) in the top surface of lamina 300. The passages which comprise the 5-branch collection manifold 50 in the exemplary apparatus are formed using an 5 etching technique.

In Figure 5 passages 160A, 160B, 160C, 160D, in combination with the branches 40-1, 44-1; 44-1, 40-2; 10 40-2, 44-2; and 40-3, 44-3 of distribution manifolds 40, 44 (Figures 4 and 7) form an array of T-mixer structures 62 whose operation will be subsequently described in conjunction with Figures 6, 7. The passages 164 in the bottom surface of the lamina 100 and the passages 264 in the top surface of lamina 200 cooperate to form an array 15 of mixing chambers 64, each of which comprises a plurality of intersecting channels (Figure 6).

At the left of Figure 5 is vertical passage 20V, which extends through lamina 100 to connect inlet port 20 with the common chamber 40M of manifold 40. At the right of Figure 5 is vertical passage 24V, which extends 20 through lamina 100 to connect inlet port 24 with common chamber 44M of manifold 44. A third branch 50-3 of 5-branch manifold 50 is formed by corresponding mirror image pathways 250-3 and 350-3 (Figure 7) in the bottom surface of lamina 200 and the top surface of lamina 300, 25 respectively. Mixing chambers 64A3, 64B3, 64C3, 64D3 are visible in Figure 5.

Figure 6 shows an arrangement of pathways that cooperate to form an array of mixer/reaction chambers 60 and a pair of distribution manifolds 40, 44. First 30 distribution manifold 40 is comprised of common chamber 40M and branch passages 40-1, 40-2, 40-3. The chamber 40M is formed by the combination of chamber 140M, on the bottom surface of the first lamina 100, and chamber 240M on the top of lamina 200. Also shown is the opening of

vertical pathway 20V which connects input port 20 with the common chamber 40M.

Second distribution manifold 44 is comprised of common chamber 44M and branch passages 44-1, 44-2, 44-3.

- 5 The chamber 44M is formed by the combination of chamber 144M, on the bottom surface of the first lamina 200, and chamber 244M on the top of lamina 200. Also shown is the opening of vertical pathway 24V which connects input port 24 with the common chamber 44M.
- 10 A series of serpentine pathways 164 are formed on the bottom surface of the first lamina 100, which cooperate with corresponding serpentine pathways 264 of lamina 200, to form the array of intersecting-channel mixing elements 64 (here, 64A5 formed from 164A5 and 264A5 in Figure 6) of the mixer array 60. The mixer array 60 comprises multiple groups 60A (e.g., 64A1-64A5), 60B, 60C, 60D of multiple parallel mixers 64. In the specific example shown, there are five mixers in each group, that are respectively designated
- 15 20 64A1, 64A2, 64A3, 64A4, 64A5 through 64D1, 64D2, 64D3, 64D4, 64D5. Figure 6 also shows chambers 40M and 44M, previously mentioned.

- 25 Each mixer 64 is comprised of two serpentine pathways, a first pathway 164 formed on the bottom of the first lamina 100 and a second pathway 264 formed on the top of the second lamina 200. The first and second pathways are each comprised of a series of straight segments and turning segments connected together to form a continuous path. The first and second pathways are
- 30 positioned on abutting surfaces with the segments longitudinally offset such that the corresponding turning segments intersect repeatedly. The mixer element 64 (as illustrated by mixer element 64A2) may be described as having a double serpentine path (best seen
- 35 in Figure 11).

Four pathways 160A, 160B, 160C and 160D, cooperate respectively with branches 40-1, 40-2, 40-3 of manifold 40, branches 44-1, 44-2, 44-3 of manifold 44 and a first straight segment 264S of each serpentine pathway 264 to 5 form a series of T-mixers 62 (best seen in Figure 7). Each first straight segment 264S thus connects each T-mixer 62 with each serpentine mixer 64 of mixer array 60.

Figure 7 shows the arrangement of pathways that 10 cooperate to form a collection manifold 50. As seen in Figures 6 and 7, collection manifold 50 is comprised of common chamber 50M and branch passages 50-1, 50-2, 50-3, 50-4, and 50-5. The vertical pathways 64A1V, 64A2V, 64A3V, 64A4V, and 64A5V connects the last segment of 15 mixer 64A1, 64A2, 64A3, 64A4, 64A5 with the collection manifold branches 50-1, 50-2, 50-3, 50-4, and 50-5, respectively; the vertical pathway 64B1V, 64B2V, 64B3V, 64B4V, and 64B5V connects the last segment of mixer 64B1, 64B2, 64B3, 64B4, 64B5 with the collection 20 manifold branches 50-1, 50-2, 50-3, 50-4, and 50-5, respectively; the vertical pathway 64C1V, 64C2V, 64C3V, 64C4V, and 64C5V connects the last segment of mixer 64C1, 64C2, 64C3, 64C4, 64C5 with the collection 25 manifold branches 50-1, 50-2, 50-3, 50-4, and 50-5, respectively; and the vertical pathway 64D1V, 64D2V, 64D3V, 64D4V, and 64D5V connects the last segment of mixer 64D1, 64D2, 64D3, 64D4, 64D5 with the collection 30 manifold branches 50-1, 50-2, 50-3, 50-4, and 50-5, respectively. Vertical passage 30V connects collection manifold 50 with the outlet port 30 on the first side of lamina 100. Vertical passage 34V connects manifold 50 with both outlet ports 34 on the first side of lamina 100 and the second side of the lamina 300, as best seen in Figure 5.

As best seen in Figure 8, each pathway 160 cooperates with manifolds 40 and 44 and a first straight segment of serpentine pathway 264 to form a T-mixer 62. This connects with a serpentine mixer 64, comprised of 5 the multiple straight segments 264S and turning segments 264T. As also may be seen in Figure 8 each portion of pathway 160, e.g., portion 160A-1 between manifold 40-1 and segment 264S1 and portion 160A-2 between manifold 44-2 and segment 264S1, may be of a different cross- 10 sectional size to provide the desired flow rate of each chemical being mixed. The number of straight and turning segments 164S, 164T and 264S, 264T and the cross-sectional size of each segment of 164 and 264 may be selected, according to the mixing requirements and 15 flow characteristics of the chemicals being processed.

As seen in Figure 9 the serpentine pathways 164, 264 are comprised of straight segments designated with a suffix letter "S" (171S, 172S, 173S, 174S, 175S, and 271S, 272S, 273S, 274S, 275S) and turning segments 20 designated with a suffix letter T (171T, 172T, 173T, 174T, 175T, and 271T, 272T, 273T, 274T, 275T). Corresponding turning segments (e.g., 171T, 271T) of pathways 164 and 264 intersect in a shearing fashion at a ninety (90) degree angle of attack. By slightly 25 offsetting the position of pathway 164 relative to pathway 264 this angle of attack may be changed if desired. By rotating lamina 100 relative to lamina 200, and suitably altering the spacing of the straight segments (suffix S) and turning segments (suffix T), 30 almost any desired angle of attack may be achieved. Angles of attack within the range of 20 degrees to 160 degrees may be readily realized, as is contemplated being within the scope of this invention.

Figure 10 shows an alternate arrangement of 35 channels, which may be used in place of the arrangement

shown in Figures 6, 8, and 9. Figure 10 shows two channels 181, 182 in the bottom surface of lamina 100 that join at a point of intersection, traverse in a combined configuration as channel 183, are subsequently 5 separated into two channels 184, 185, and then recombined as channel 186, separate into channels 187, 188. Channel 281 in the top surface of lamina 200 separates at a point of intersection into two channels 282, 283, which are then recombined as channel 284, are 10 subsequently separated into two channels 285, 286, and then recombined as channel 287. Channel 181 intersects with channel 282 and channel 182 intersects with channel 283 at predetermined angles of attack and shear rates. Similarly channel 184 intersects with channel 282, 15 channel 185 intersects with channel 283, channel 184 intersects with channel 286, channel 185 intersects with channel 287, channel 187 intersects with channel 286, channel 188 intersects with channel 287.

Figure 11 depicts the channels of Figure 9 in a 20 somewhat simplified manner to better visualize the shape, relative position and intersections of the channels. As may be appreciated from Figures 9 and 11, two channels repeatedly intersect to form a double helix configuration, wherein a first channel 164 is comprised 25 of straight segments and turning segments in a first plane, a second channel 264 is comprised of straight segments and turning segments in a second plane.

Chemicals within a first channel are repeatedly subjected to the sequence of: (a) a turbulent left hand turn, (b) a turbulent left hand shearing intersection with chemicals in a second channel, (c) a turbulent right hand turn, (d) a turbulent right hand turn, (e) a turbulent right hand shearing intersection with chemicals in the second channel, (f) a turbulent left 35 hand turn.

Figure 12 depicts the channels of Figure 10 in a somewhat simplified manner to better visualize the shape, relative position and intersections of the channels. In this figure it can be seen that the two channels intersect at angles of attack and shear rates. Each channel comprises two or more channels that join at a point of intersection, traverse in a combined configuration, are subsequently separated into two or more channels, and then subsequently rejoin.

Figure 13(a)-(c) is a plan view of the channels of Figure 9. It shows channel 164 alone (Figure 13(c)), channel 264 alone (Figure 13(a)), and 164 superimposed with 264 (Figure 13(b)). At the point of intersection of the channels a centerline 164C of channel 164 and a centerline 264C of channel 264 are shown. The included angle α between these centerlines is defined as the "angle of attack" of the two channels.

Multiple units of the chemical mixing and reaction apparatus can be used in sequence or in tandem. The incorporation of units in any number of sequential or tandem patterns is a design choice to be made by those skilled in the art and according to the desired chemical processing result.

As seen in Figures 9 and 10 the particular angles of the channel walls produced at the turning sections (e.g., 171T) are due to the orientation of the crystal planes of the silicon. In the illustrated example each lamina comprises a silicon wafer which has the major planar surface of the wafer in the {100} crystal plane. The sides of the straight segments (e.g. 171S, 271S) are aligned to be in the {110} crystal planes. When etched with an anisotropic etch, the major sides of the segments are bevelled from the vertical at a 57 degree angle and are in the {111} crystal planes. The outside corners between the straight segment 171S and the

turning segment 171T, are not sharp right angles but are beveled by two so-called beveling faceting planes, which are the {210} crystal planes, thus producing the bevelled corners which are best seen in Figures 9 and 5 10.

FABRICATION OF THE APPARATUS

The apparatus of the present invention is achieved by a multi-step fabrication process. First, a series of planar laminae or wafers, are processed to form desired 10 patterns of pathways on one or both major surfaces of each lamina or through the thickness of the lamina. The laminae are then joined together to form a plurality of intersecting channels that accommodate the passage of chemicals.

15 In the preferred embodiment of the figures, the laminae are arranged and precisely oriented to locate the channels in adjacent laminae. These channels may be continuous or discontinuous along each of the laminae. Discontinuous channels are continuously aligned between 20 adjacent laminae sufficient to form a continuous pathway therethrough. A typical channel is comprised of straight segments and turning segments in a first plane, straight segments and turning segments in a second plane and transition segments between the two planes. The 25 straight or turning segments of a channel may be formed as a groove or trough entirely within one lamina. When assembled this groove cooperates with the facing planar surface of the adjacent lamina to close the cross-section of the channel. This is best illustrated by the 30 cross-sectional view of distribution manifold 40 (seen as 40-1, 40-2, and 40-3) in Figure 4. Mirror image grooves or troughs on facing surfaces of adjacent laminae may cooperate to form a channel having a symmetrical cross-section. This is best illustrated by 35 the cross-sectional view of collection manifold 50 (seen

as 50-1, 50-2, 50-3, 50-4, and 50-5) in Figure 4. The transition segments of the channels may be passages that pass through a lamina to connect a channel segment on a first lamina with another segment on a second lamina or 5 with a distribution or collection manifold. This is best illustrated by the cross-sectional view of vertical pathways 64A3V through 64D3V in Figure 5.

Processing of Laminae

The processing of the laminae to form pathways may 10 be performed by a procedure selected from the group of: subtractive processes, additive processes, and forming processes. Subtractive processes comprise chemical etching, electrochemical machining (ECM), electrical discharge machining (EDM), laser ablation, drilling and 15 cutting, abrasive grinding and single diamond point cutting (such as used to fabricate ceramic parts). Additive processes comprise deposition processes, such as electroforming, selective plating, chemical vapor deposition, stereo lithographic photoforming, and 20 welding. Forming processes comprise molding, casting, and stamping. Wear resistant coatings, such as metalloid carbides, in the form of thin films, may be optionally deposited on the processed laminae before bonding.

25 Materials of Construction

Selection of lamina materials is based upon the material's compatibility with the chemicals to be mixed and reacted. As used herein "compatibility with the chemicals to be mixed and reacted" includes without 30 limitation: resistance to chemical degradation; operating conditions, such as temperature and pressure; thermal conduction requirements; required features to be created in the lamina, including size, geometrical shape and precision; the sealability of the lamina material; 35 and economic considerations.

A variety of materials, may be used to construct the mixing/reaction units of the present invention. Crystalline materials that can be processed using photolithographic techniques may be used, especially 5 when a mixing/reaction unit with extremely small cross-sectional passages are desired, for instance when gasses that react rapidly are to be processed. Such crystalline materials would typically include elements from groups III through V of the periodic table, for 10 example silicon. Other compound materials, selected for their resistance to chemical attack, may be used to fabricate the laminae or may be used as coatings on the laminae. Ceramic materials may be used, such as silicon carbide, tungsten carbide, alumina and sapphire for 15 instance using known molding, pressing and sintering techniques, to form the laminae. Thin films may be deposited on the surfaces of the laminae, such as by chemical vapor deposition techniques, to improve resistance of the passages to chemical attack or to 20 facilitate bonding of the laminae. Glass materials, such as fused quartz, pure silica glass and borosilicate glass, as well as composite materials, such as ceramic composites and polymeric composites may be used.

For example, wafers similar to those used to 25 fabricate semiconductor electronics components, such as single crystal silicon wafers, may be used. For such silicon wafers a combination of subtractive techniques may be used to form the passages. The laminae are subsequently stacked in precise alignment and joined 30 together (as by bonding or clamping) into an integral structure. As illustrated in the example of Figure 1, three laminae are joined as an apparatus. The laminae may be of identical or different materials. The outer laminae may be silicon or a protective material such as 35 metal, ceramic, composite material or glass while the

inner lamina would typically be silicon. If all the laminae are silicon, thermal fusion bonding is a preferred method of joining the laminae because the strength of the bond achieved approaches that of the 5 laminae themselves.

OPERATION OF THE APPARATUS

Having reference to Figures 1-3, the two materials to be reacted flow into the unit through input ports 20, 24, through vertical passages 20V, 24V into distribution 10 manifolds 40, 44 and into mixer array 60. Optional temperature control means (not shown) may be used to maintain the mixer array 60 at the desired temperature. The mixed material is collected in collection manifold 50 and passed through vertical passage 30V to the outlet 15 port 30 or through vertical passage 34V to the outlet port 34.

Additional microfabricated process control elements, such as proportional valves, pressure, temperature and flow sensors, may be incorporated with 20 the structure of the present invention. These elements, when used with external controls could regulate the flow of reactants within the integrated chemical processing unit and thus control the residence time. Any rapid chemical process, such as hydrolysis, nitration, 25 polymerization, and oxidation may be practiced using the integrated structure of the present invention.

FABRICATION OF THE LAMINAE

In the preferred embodiment, most steps of the fabrication process generally correspond to known 30 semiconductor processing techniques for silicon wafers. The photo-tools for the patterns for each side of each wafer are prepared using well known computer-aided-design techniques. Already polished silicon wafers, having the {100} crystal plane and other orientations on 35 the major surfaces may be purchased from commercial

sources. The polished wafers are first cleaned using a well-known general cleaning technique, such as the "RCA process". An oxide film is grown on the wafer using well-known standard techniques. A nitride layer is 5 deposited over the oxide layer using a known chemical vapor deposition method. The nitride layer protects the oxide layer from attack by the etch used to etch the silicon. A photoresist is applied, following the directions of the photo resist manufacturer, using the 10 well-known spin coating technique.

The desired pattern is formed by first masking the wafer with a photo-tool having an image of the desired pattern, which is precisely aligned with the crystal planes of the wafer. Straight portions of the pattern 15 are typically aligned along the {110} crystal plane. After exposing and developing the photoresist, the undeveloped photoresist is stripped to expose part of the nitride/oxide film layer. The exposed nitride/oxide film is finally etched to form an nitride/oxide film 20 negative image of the desired pattern.

The pathways are formed in the surfaces of the wafers by etching the silicon, using either isotropic or anisotropic etch, the choice of which is dependent on the shape of the pathway desired. Curved shapes are 25 etched using an isotropic etch. Straight shapes may employ either etch, depending on the desired cross-sectional shape pathway. If a trapezoidal cross-section is desired an anisotropic etch is used.

If a given wafer is to be etched on both major 30 surfaces using the same etch, both sides of the wafer may be masked with resist, the resist exposed with the desired pattern on each surface, developed, washed and the nitride/oxide etched simultaneously on both surfaces. Then the silicon may be simultaneously etched 35 on both surfaces. If different types of etchants are to

be used on each side of the wafer, all steps for the first etch are completed and then the steps are repeated for the second etch. After all the etching steps have been completed the vertical passages or vias through the 5 wafer are formed by laser cutting through the wafers, typically using a pulsed neodymium-YAG laser cutting system. After laser cutting, the wafers are again recleaned to remove cutting debris. The remaining nitride layer of the negative image is removed from the 10 wafer, by using a suitable solvent, such as boiling phosphoric acid, exposing the undamaged oxide layer. The remaining oxide layer negative image may optionally be removed from the wafer, by using a suitable solvent, such as buffered hydrogen fluoride. The wafer is 15 recleaned, using the technique as described above.

Other techniques may be used which are dependent upon the lamina material. Laminae which are comprised of group III, IV or V material are processed using etching, grinding, drilling and polishing techniques. 20 Laminae comprised of glass, Pyrex, or fused silica are fabricated using conventional glass cutting, drilling, grinding, and polishing techniques. Laminae comprised of ceramic materials may be formed by slip casting, consolidated by pressing and fired using well known 25 techniques.

When all the laminae have been individually processed the laminae are carefully stacked in a precisely aligned manner and fusion bonded. To achieve good bonding of silicon laminae, the surfaces should be 30 highly planar and the oxide layers on each surface should be undamaged. Since silicon is somewhat transparent in the infrared, a microscope with an infrared video camera may be used, with optional alignment indicia on each wafer, to insure precise 35 alignment of the wafers before they are fusion bonded.

If an outer lamina is comprised of glass, this lamina is then anodically bonded to the already fused stack of inner laminae.

Those skilled in the art, having the benefit of the 5 teachings of the present invention as hereinabove set forth, can effect numerous modifications thereto. It is readily appreciated that such modifications can be made without departing from the spirit of the scope of the present invention. Accordingly, such modifications are 10 to be construed as being encompassed within the scope of the present invention as set forth in the appended claims.

CLAIMS

1. An apparatus for use in an integral structure for chemical mixing and reacting of one or more chemicals, comprising a plurality of laminae joined together and having a plurality of intersecting channels formed therethrough which accommodate the passage of one or more chemicals,
 - 5 said laminae comprising a material selected for compatibility with the one or more chemicals,
 - 10 said channels being formed so that chemicals are combined at intersections therein at sufficient angles of attack and shear rates so that chemical reactions are not limited by mass transfer considerations.
- 15 2. The apparatus of Claim 1 wherein the channels are generally planar, a first channel being disposed in a first plane and a second channel being disposed in a second plane, wherein the first and second planes are parallel and adjacent to each other, and the channels
- 20 30 are generally serpentine and are offset from each other such that the first and second channels repeatedly intersect in a shearing fashion and form angles of attack at points of intersection.
- 25 4. The apparatus of Claim 3 wherein two or more channels are positioned at a point of intersection to form an angle of attack of 20 to 160 degrees.
5. The apparatus of Claim 3 wherein the angle of attack is 90 degrees.
- 30 6. The apparatus of Claim 1 wherein shear rate is sufficient to disrupt boundary layer formation.
7. The apparatus of Claim 1 wherein two or more channels intersect at aforesaid angles of attack and
- 35 35 shear rates, each channel comprising two or more

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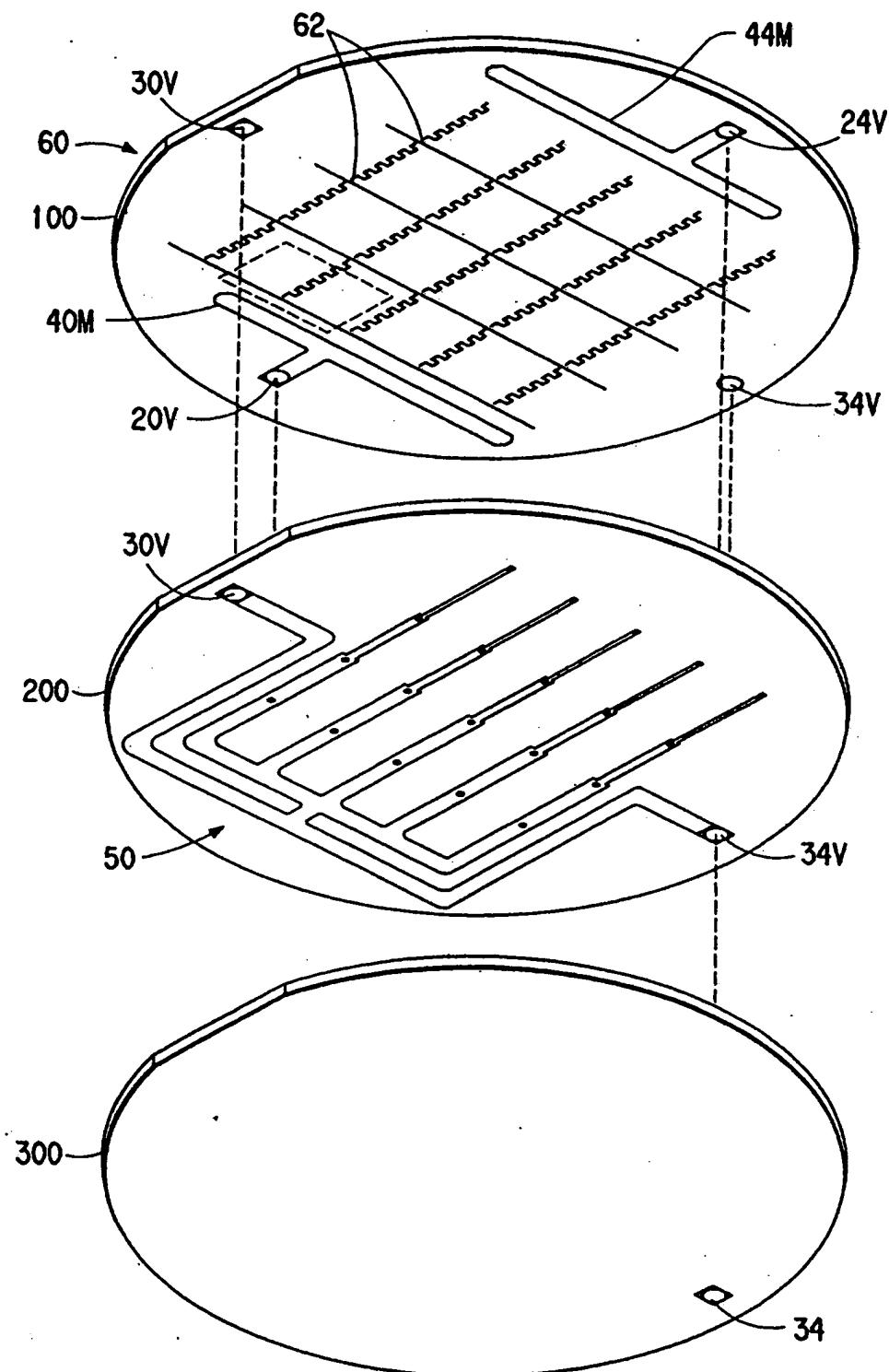
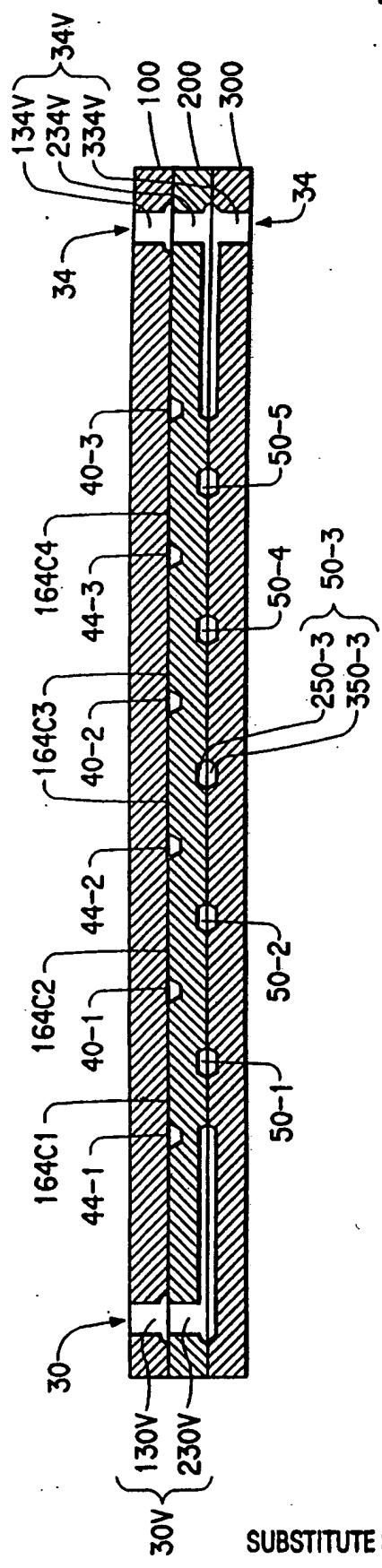


FIG.3

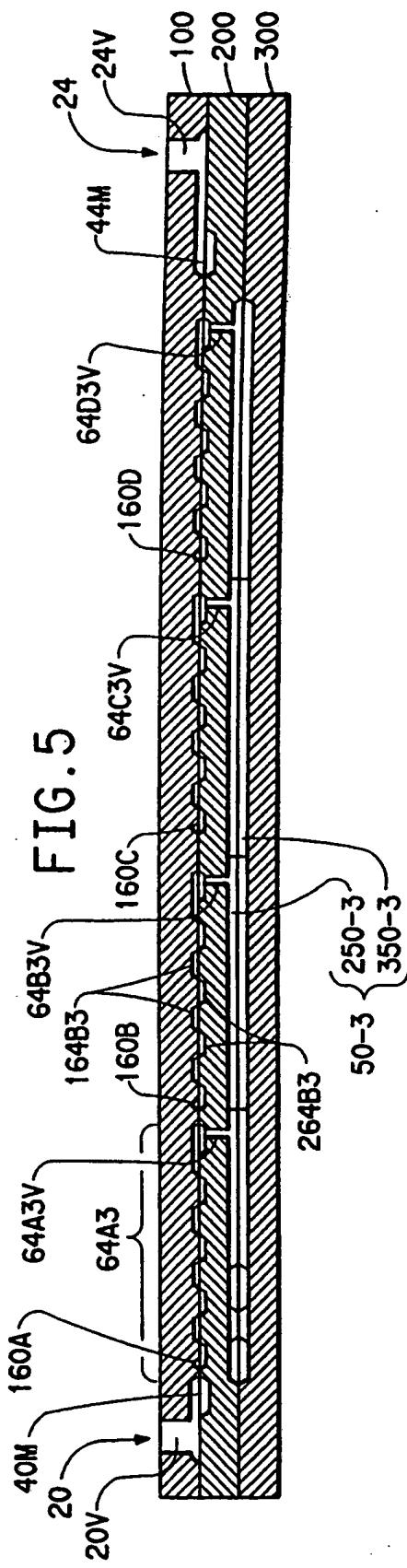
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FIG. 4



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FIG. 5



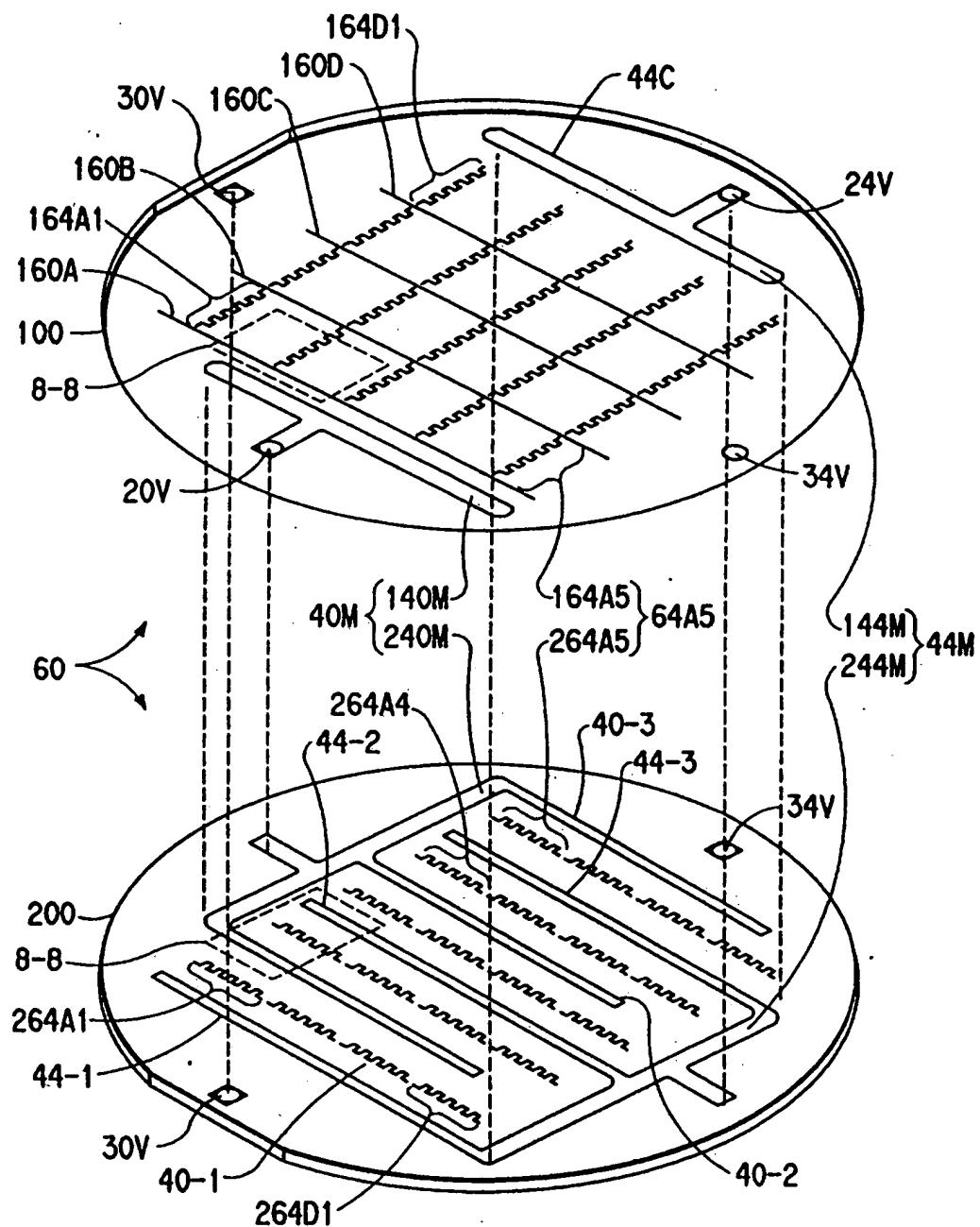


FIG. 6

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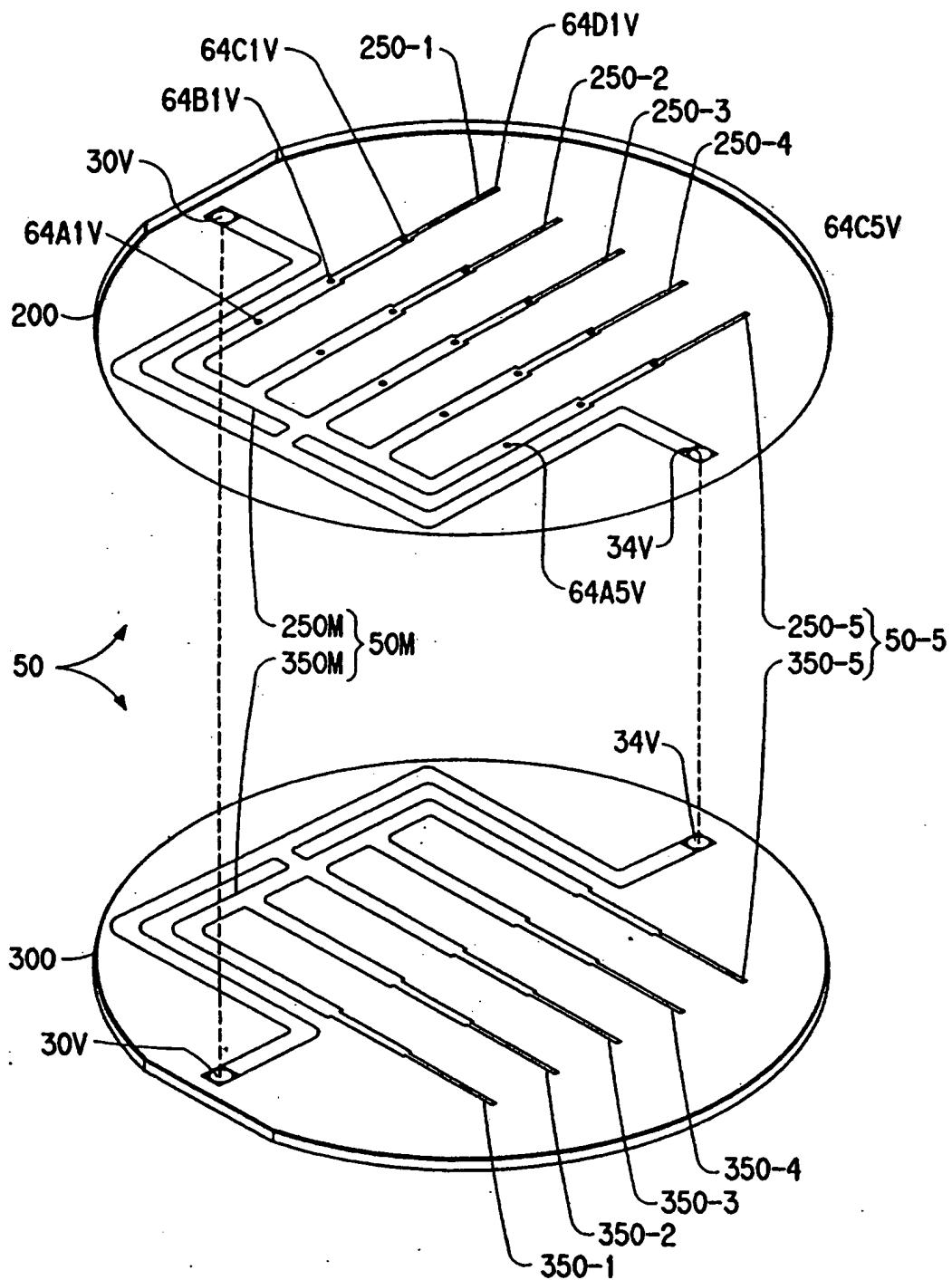


FIG. 7

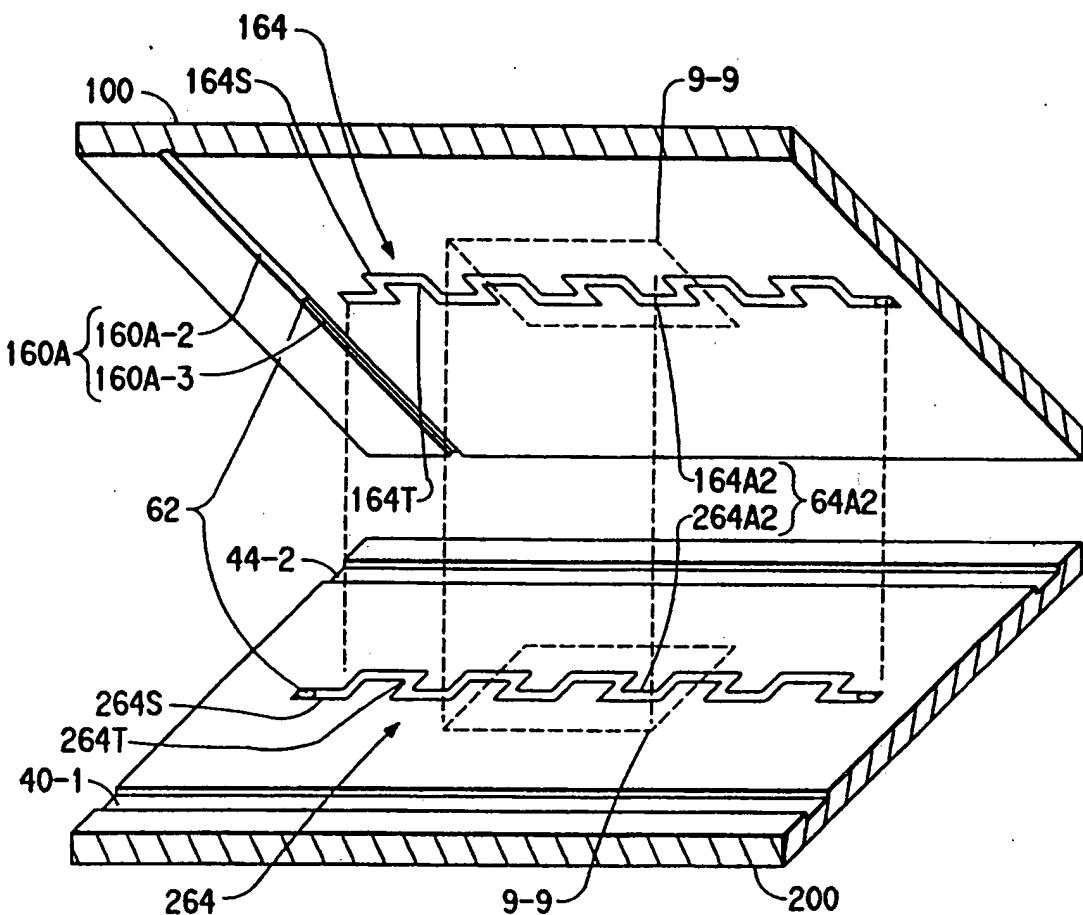


FIG. 8

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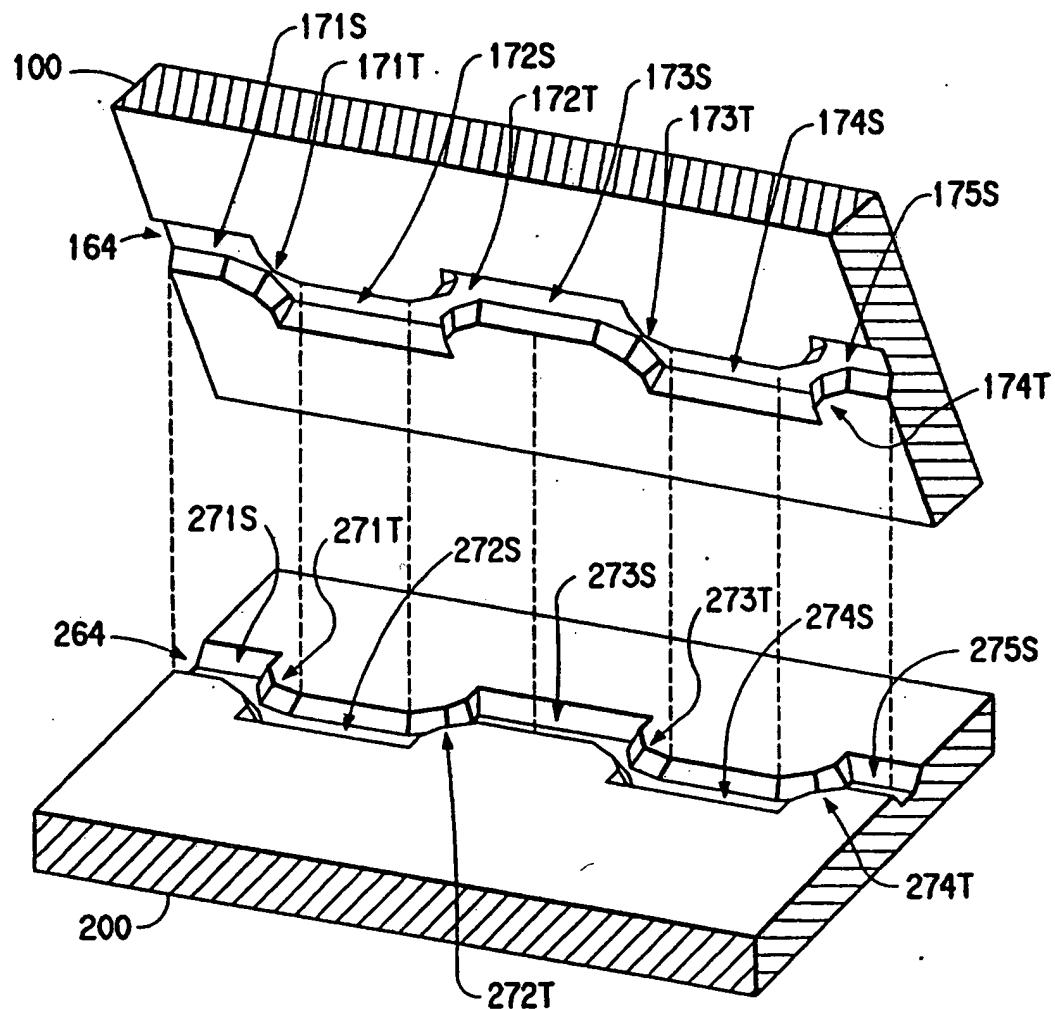


FIG. 9

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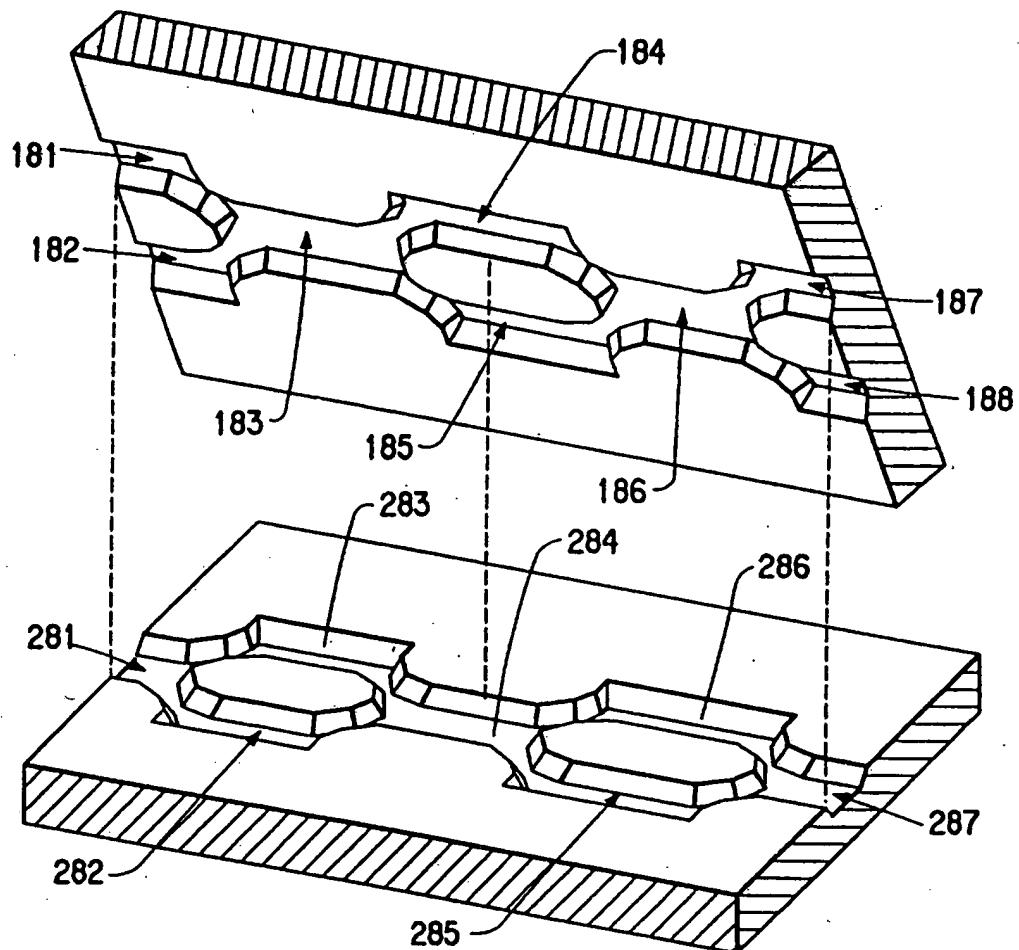


FIG.10

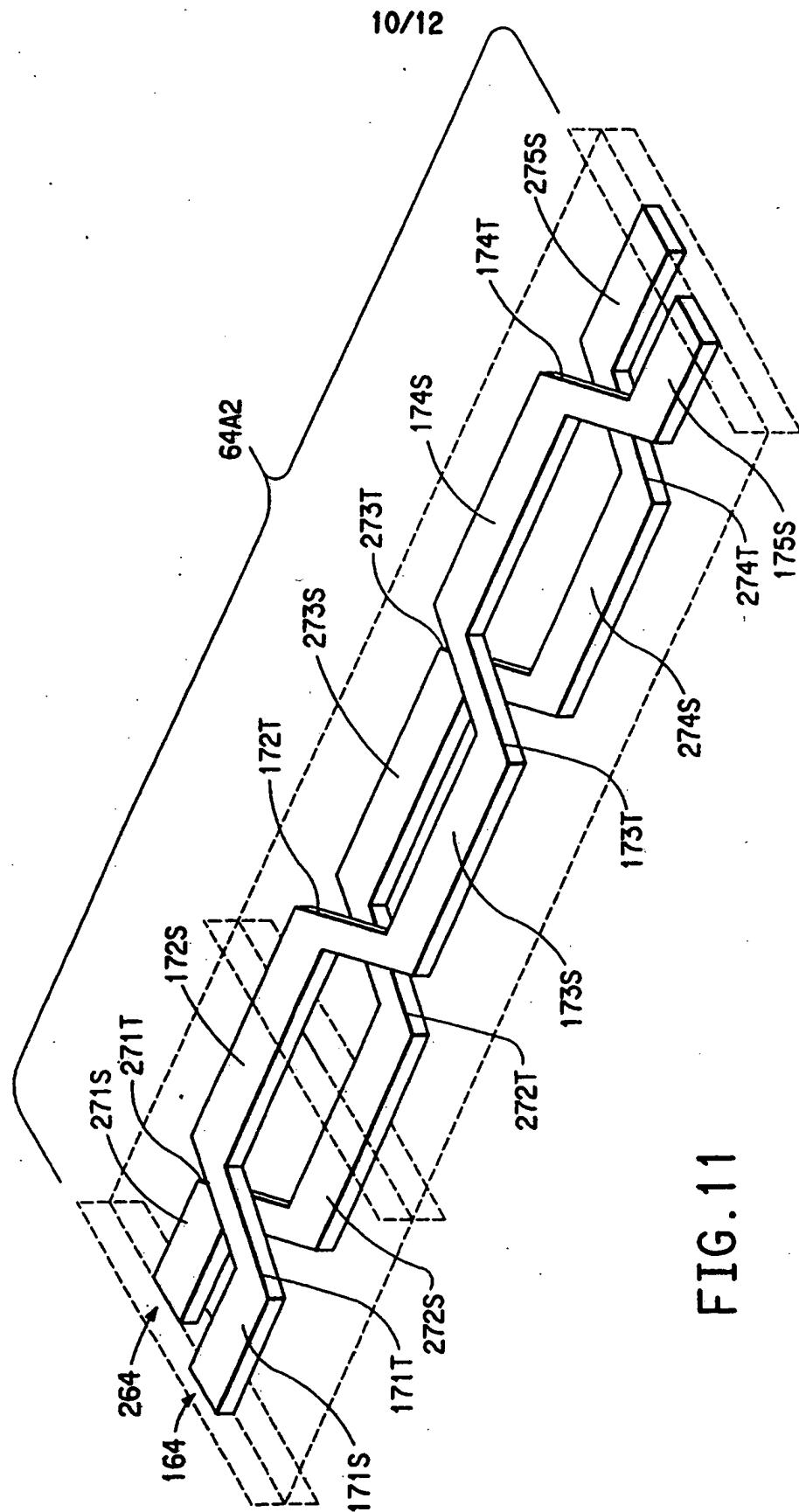


FIG. 11

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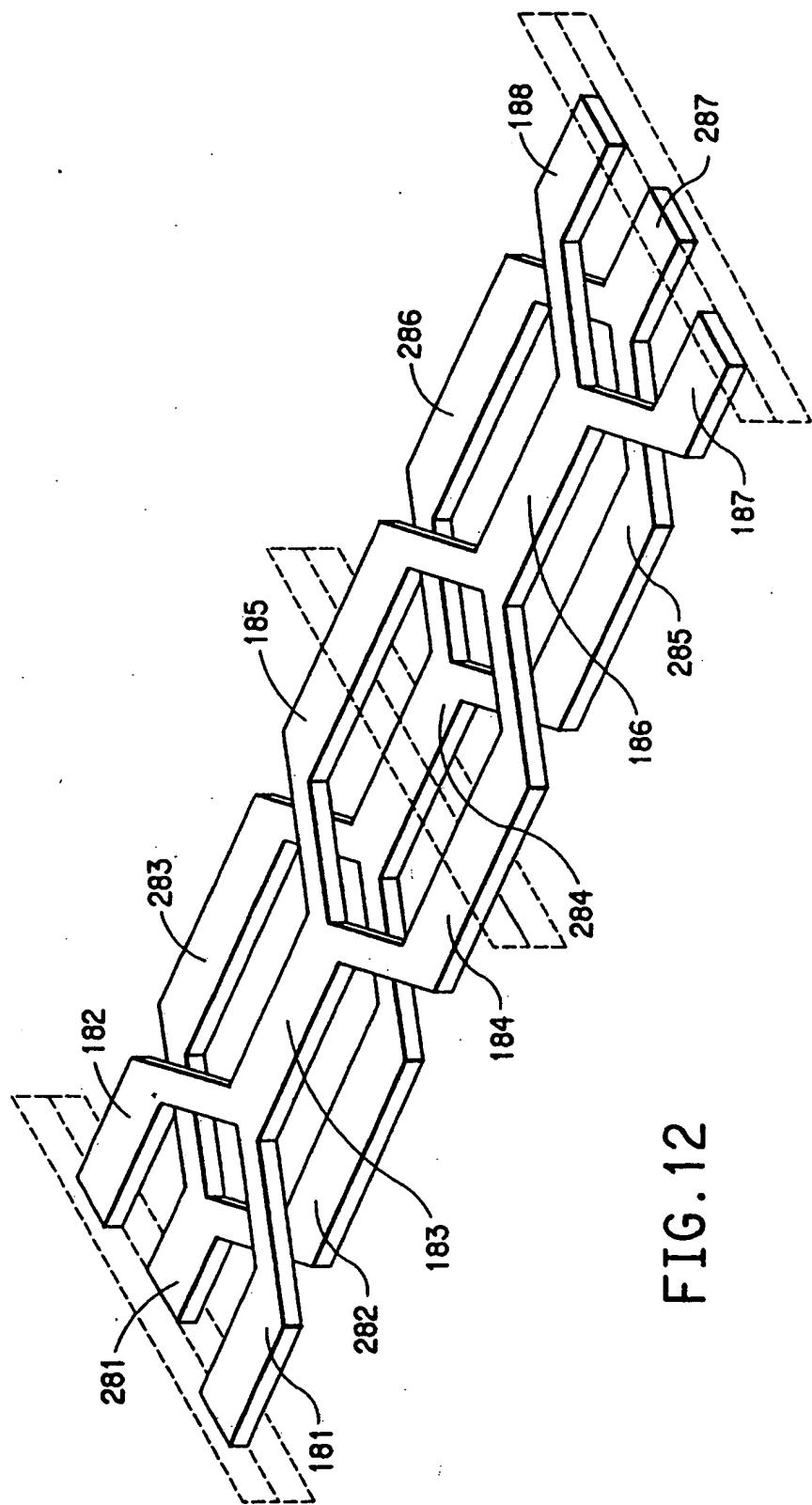


FIG. 12

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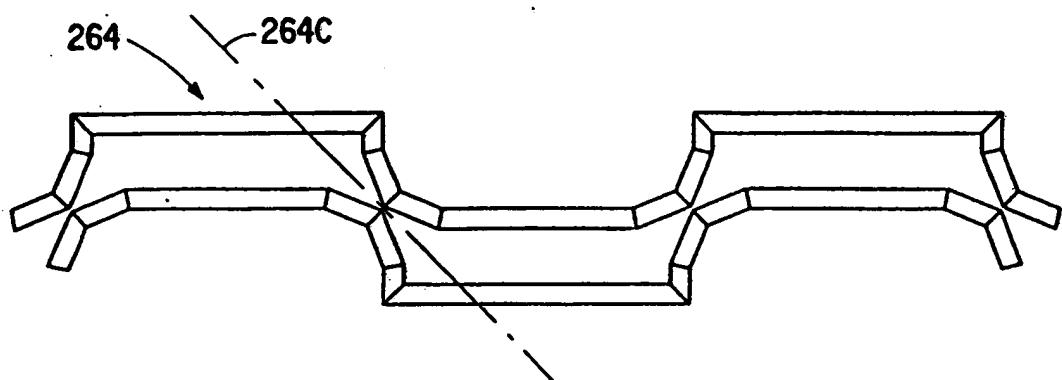


FIG. 13A

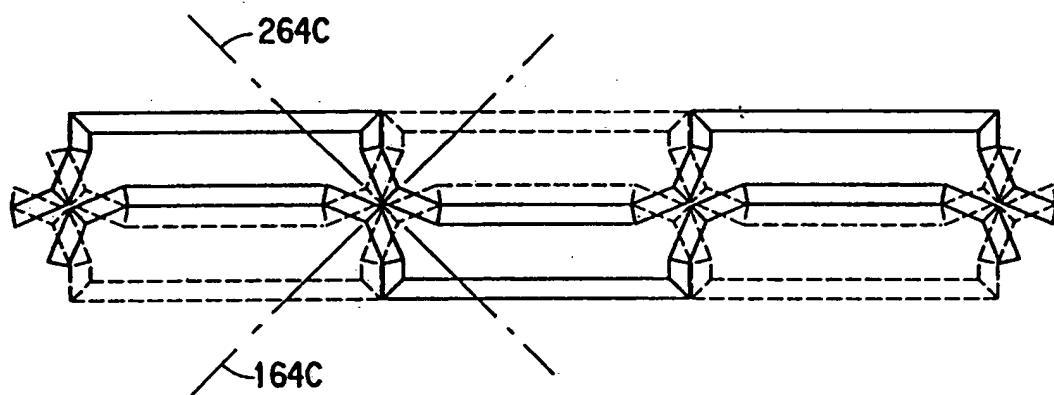


FIG. 13B

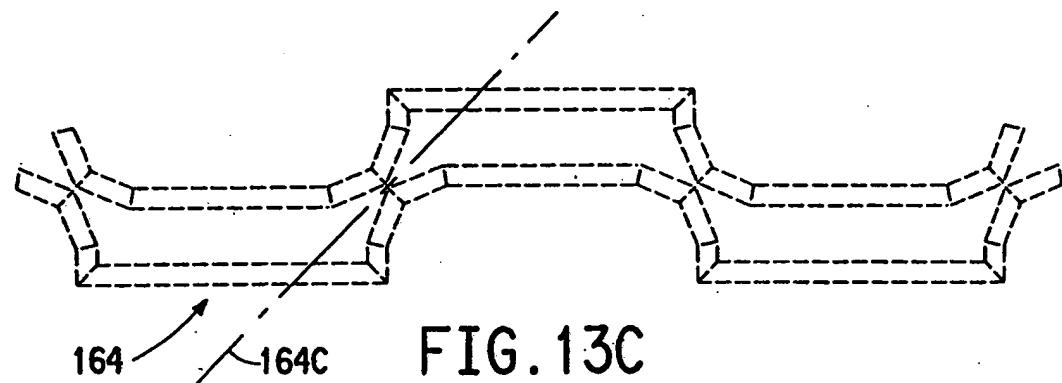


FIG. 13C

INTERNATIONAL SEARCH REPORT

Inte onal Application No
PCT/US 95/09834

| | |
|--|--|
| A. CLASSIFICATION OF SUBJECT MATTER | |
| IPC 6 | B01J19/00 B01F5/06 B01J12/00 B01J14/00 B01J19/12 |
| | B01J35/04 B01L3/00 |

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 B01J B01F B01L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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| A | FR,A,2 640 620 (INSTITUT FRANÇAIS DU PÉTROLE) 22 June 1990 see abstract see page 1, paragraph 1 see page 2, paragraph 2 - page 4, paragraph 2 see page 9, paragraph 5 - page 10, paragraph 3 see figures 1-3 --- | 1 -/- |

Further documents are listed in the continuation of box C.

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Date of the actual completion of the international search

27 November 1995

Date of mailing of the international search report

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C(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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| A | US,A,3 856 270 (FMC CORPORATION) 24 December 1974 see abstract see figures 1-4 see column 3, line 8 - column 4, line 35 --- | 1 |
| A | EP,A,0 496 521 (HITACHI, LTD.) 29 July 1992 see the whole document --- | 1 |
| A | US,A,5 320 139 (MILLIPORE CORPORATION) 14 June 1994 see abstract see column 4, line 5 - column 7, line 29 see figures --- | 1 |
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International Application No
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| US-A-5320139 | 14-06-94 | DE-T- | 622115 | 28-09-95 |
| | | EP-A- | 0622115 | 02-11-94 |
| | | JP-A- | 7068154 | 14-03-95 |



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|--|--|--|---|
| (51) Internationale Patentklassifikation 6 : B01F 5/02, 5/06, B01J 19/24 | | A1 | (11) Internationale Veröffentlichungsnummer: WO 97/16239 (43) Internationales Veröffentlichungsdatum: 9. Mai 1997 (09.05.97) |
| <p>(21) Internationales Aktenzeichen: PCT/EP96/03162</p> <p>(22) Internationales Anmeldedatum: 18. Juli 1996 (18.07.96)</p> <p>(30) Prioritätsdaten: 195 40 292.8 28. Oktober 1995 (28.10.95) DE</p> <p>(71) Anmelder (für alle Bestimmungsstaaten ausser US): FORSCHUNGSZENTRUM KARLSRUHE GMBH [DE/DE]; Weberstrasse 5, D-76133 Karlsruhe (DE). BAYER AG [DE/DE]; D-51368 Leverkusen (DE).</p> <p>(72) Erfinder; und (75) Erfinder/Anmelder (nur für US): SCHUBERT, Klaus [DE/DE]; Geigersbergstrasse 54, D-76227 Karlsruhe (DE). BIER, Wilhelm [DE/DE]; Grabener Weg 10, D-76344 Eggenstein-Leopoldshafen (DE). HERRMANN, Erhard [DE/DE]; Felix-von-Roll-Strasse 11, D-51375 Leverkusen (DE). MENZEL, Thomas [DE/DE]; Richrather Strasse 248, D-40723 Hilden (DE). LINDER, Gerd [DE/DE]; Stephan-Lochner-Weg 2, D-76149 Karlsruhe (DE).</p> <p>(74) Anwalt: RÜCKERT, Friedrich; Forschungszentrum Karlsruhe GmbH, Stabsabteilung Patente und Lizizenzen, Weberstrasse 5, D-76133 Karlsruhe (DE).</p> | | <p>(81) Bestimmungsstaaten: CA, CN, JP, KR, US, europäisches Patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).</p> <p>Veröffentlicht Mit internationalem Recherchenbericht.</p> | |
| <p>(54) Title: STATIC MICROMIXER</p> <p>(54) Bezeichnung: STATISCHER MIKROVERMISCHER</p> <p>(57) Abstract</p> <p>Disclosed is a static micromixer with a mixing chamber and a duct component upstream of the former for separate feeding to the mixing chamber of the fluids to be mixed or dispersed. The aim of the invention is to design the duct component in a static mixer of this generic type in such a way as to improve the efficiency of the mixing process in the mixing chamber. The invention is characterised by the following features: a) the wafer-like elements A and B for the duct component each consist of thin films with a thickness of 10-1000 microns, preferably < 250 microns, the lateral dimension being of the order of millimetres, a family of adjacent grooves being formed in each wafer-like element such that when the foils are superposed one on top of the other a separate series of channels is formed for feeding the fluids A' and B' to be mixed; b) the grooves have depths of less than 1000 microns, preferably less than 250 microns, widths of between 1 micron and the millimetre range, but preferably less than 1000 microns, the wall thicknesses of the intermediate ridges and groove floors being less than 1000 microns and preferably less than 250 microns; c) each family of grooves in the stacked films follows an arc and lead alternately from the mixing chamber to a feed chamber for the fluid A' or B' in such a way that all families of grooves in the films are aligned parallel to one another and discharge into the mixing chamber.</p> <p>(57) Zusammenfassung</p> <p>Die Erfindung betrifft einen statischen Mikrovermischer mit einer Mischkammer und einem vorgeschalteten Führungsbauteil für die getrennte Zufuhr von zu mischenden oder zu dispergierenden Fluide zu der Mischkammer. Die Erfindung hat zur Aufgabe, bei einem statischen Vermischer der gattungsgemäßen Bauart das Führungsbauteil für die getrennte Zufuhr der zu mischenden Fluide zur Mischkammer so zu gestalten, daß eine erhöhte örtliche und zeitliche Effektivität der Vermischung in der Mischkammer erzielt wird. Die Erfindung ist durch die folgenden Merkmale gekennzeichnet: a) die plattenartigen Elemente A und B für das Führungsbauteil bestehen jeweils aus dünnen Folien mit einer Dicke von 10 bis 1000 µm, vorzugsweise < 250 µm, bei einer lateralen Ausdehnung im Millimeterbereich, in die je eine Schar benachbarter Nuten eingearbeitet ist, so daß beim Übereinanderschichten der Folien je eine Reihe von Kanälen für die Führung der zu mischenden Fluide A' und B' entsteht; b) die Nuten haben Tiefen von < 1000 µm, vorzugsweise < 250 µm, Breiten von 1 µm bis in den Millimeterbereich, vorzugsweise jedoch < 1000 µm, bei Wanddicken der Zwischenstege und Nutböden von < 1000 µm, vorzugsweise < 250 µm; c) die jeweiligen Scharen von Nuten in den Übereinandergeschichteten Folien verlaufen jeweils bogenförmig gekrümmmt und abwechselnd von der Mischkammer zu je einer Zufuhrkammer für das Fluid A' bzw. B' in der Weise, daß alle Scharen von Nuten der Folien parallel zueinander ausgerichtet in die Mischkammer ausmünden.</p> | | | |

LEDIGLICH ZUR INFORMATION

Codes zur Identifizierung von PCT-Vertragsstaaten auf den Kopfbögen der Schriften, die internationale Anmeldungen gemäss dem PCT veröffentlichen.

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Statischer Mikrovermischer

Die Erfindung betrifft einen statischen Mikrovermischer mit einer Mischkammer und einem vorgeschalteten Führungsbauteil für die getrennte Zufuhr von zu mischenden oder zu dispergierenden Fluiden zu der Mischkammer gemäß dem Oberbegriff von Patentanspruch 1.

Aus der WO91/16970 A1 (PCT/CH91/00109 v. 14.11.1991) ist eine Mischeranordnung in einer Kolonne mit Misch-, Katalysator- bzw. Kanalelementen bekannt, die in Schichten oder Lagen angeordnet sein können. Benachbarte Elemente innerhalb einer Lage oder Schicht sind gegeneinander zur Hauptströmungsrichtung geneigt angeordnet. Hierzu können die Elemente die Form eines schiefen Prismas haben, das von im Querschnitt quadratischen, gerade und parallel zueinander verlaufenden Kanälen durchzogen ist und das durch Zusammenfügen strukturierter Platten gefertigt werden kann. Bei einer so getroffenen Anordnung wird ein gleichmäßiger Strömungswiderstand bezüglich der zu mischenden Fluide erzielt, die Mischungseffektivität fällt jedoch vom Zentrum zu den Randzonen hin des als Mischkammer dienenden Kolonnenraumes am abströmseitigen Ende der Elemente stark ab.

Aus der DE 31 14 195 C2 ist eine gattungsgemäße Mischvorrichtung zur Vermischung strömender Medien mit mindestens zwei Einlaßkanälen, mit einem sich an diese anschließenden Kanalkörper sowie mit einem an die gemeinsame Auslaßseite des Kanalkörpers angeschlossenen Auslaßkanal bekannt. Der von Einzelkanälen durchzogene Kanalkörper, der zur getrennten Zufuhr der zu mischenden Medien zum Auslaßkanal dient, ist aus aufeinander aufgeschichteten Einzelwell-Einheiten aufgebaut, die aus je einer Trennwand und einem Wellbogen bestehen. Die Einzelkanäle jeder Einheit werden von den Wellungen des Wellbogens und der zugehörigen Trennwand gebildet. Die Einheiten sind so aufeinander geschichtet, daß die Einzelkanäle benachbarter Einheiten vorbestimmte Winkel zueinander bilden. In einem Ausführungsbeispiel ist die Anordnung so getroffen, daß

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der Kanalkörper gleich lange, parallele Einzelkanäle hat, wobei die Einzelkanäle der jeweiligen Einheiten abwechselnd an je einen von zwei Einlaßkanälen angeschlossen sind, auf der Auslaßseite des Kanalkörpers mit ihren Mündungen jedoch übereinander liegen. Da das jeweilige Medium nur in jeden zweiten Einzelkanal, in der Höhenerstreckung des Kanalkörpers gesehen, einströmen darf, muß der Einlaß zu der jeweils dazwischenliegenden Einheit geschlossen sein (s. Spalte 6, Zeilen 26 bis 33 in Verbindung mit Fig. 7a, 7b), wodurch Totvolumina entstehen. Aber auch bei dieser Bauart ergibt sich nur im Zentrum der Auslaßseite des Kanalkörpers eine optimale Durchmischung; zu den Randzonen hin nimmt die Mischungseffektivität ab. Auch ist das Ausformen oder Anbringen der erforderlichen Einlaß-Verschlüsse aufwendig und stößt mit kleiner werdenden Kanalquerschnitten auf fertigungstechnische Schwierigkeiten.

Ausgehend von dem vorstehend erörterten Stand der Technik hat die Erfindung zur Aufgabe, bei einem statischen Vermischer der gattungsgemäßen Bauart das Führungsbauteil für die getrennte Zufuhr der zu mischenden Fluide zur Mischkammer so zu gestalten, daß eine erhöhte örtliche und zeitliche Effektivität der Vermischung in der Mischkammer erzielt wird. Darüber hinaus soll der Vermischer hinsichtlich der Kanäle in dem Führungsbauteil eine vakuumdichte und druckfeste Ausführung ermöglichen, so daß er insbesondere in der chemischen Technik eingesetzt werden kann.

Diese Aufgabe wird mit den kennzeichnenden Merkmalen von Patentanspruch 1 gelöst. Die hierauf bezogenen Unteransprüche beinhalten vorteilhafte Weiterbildungen dieser Lösung.

Durch die bogenförmig gestalteten, parallel zueinander und zur Hauptströmungsrichtung in die Mischkammer ausmündenden Kanäle der Elemente A und B des Führungsbauteils wird über dem gesamten Ausströmquerschnitt eine gleichmäßig hohe Vermischung bei Vermeidung von Totvolumina im Führungsbauteil erzielt. Aufgrund der Mikrostrukturierung der Kanalreihen werden die zu

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vermischenden oder zu dispergierenden Fluide in eine Vielzahl feinster benachbarter Stromfäden oder Lamellen unterteilt, die beim Eintritt in die Mischkammer sich auf schnellstem und kürzestem Wege durchmischen können oder in eine Tropfen oder Blasen bildende Dispersphase und eine die kontinuierliche Phase bildende Hüllphase zerteilt werden. Die Dichte der Kanalmündungen und damit der Stromfäden am Eintritt in die Mischkammer beträgt einige tausend Mündungen bzw. Stromfäden pro cm².

Ein Ausführungsbeispiel des erfindungsgemäßen Mikrovermischers sowie ein Verfahren zur Herstellung desselben werden im folgenden anhand der Zeichnungen erläutert.

Die Figur 1 zeigt perspektivisch und in starker Vergrößerung zwei übereinander zu schichtende, genutete Folien und eine Deckplatte;

die Figur 2 zeigt den Mikrovermischer schematisch in der Draufsicht;

die Figur 3 veranschaulicht das Herstellungsverfahren für die genuteten Folien.

Gemäß Fig. 1 bestehen die plattenförmigen, übereinanderzuschichtenden Elemente abwechselnd jeweils aus dünnen Folien 1A und 1B mit einer Dicke von 30 bis 1000 µm, vorzugsweise < 250 µm bei einer lateralen Ausdehnung im Millimeterbereich. In die Folien 1A, 1B ist je eine Schar eng benachbarter, bogenförmig gekrümmter Nuten 2A, 2B eingearbeitet, so daß beim Übereinanderschichten der Folien 1A, 1B abwechselnd je eine Reihe von Kanälen 3A und 3B entsteht (siehe Fig. 2).

Die Nuten haben Tiefen von < 1000 µm, vorzugsweise < 250 µm, Breiten von 10 µm bis in den Millimeterbereich, vorzugsweise jedoch < 500 µm, bei Wanddicken der Zwischenstege 4A, 4B und Nutböden 5A, 5B von < 1000 µm, vorzugsweise < 250 µm. Die

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Nutenschar 2B verläuft von der linken, hinteren Stirnfläche 6B bogenförmig zum mittleren Bereich 6C der vorderen Stirnfläche 8B der Folie 1B, die Nutenschar 2A verläuft bogenförmig von der rechten, hinteren Stirnfläche 6A zum mittleren Bereich 6C der vorderen Stirnfläche 8A der Folie 1A, wobei beide Folienarten 1A und 1B den gleichen Grundriß haben. Dieser Grundriß hat die geometrische Form eines Fünfecks, gebildet aus einem Rechteck mit hieran angeschlossenem, gleichschenkligem Dreieck, wobei eine Seite des Rechtecks die Grundseite des gleichschenklichen Dreiecks bildet.

Schichtet man eine Vielzahl dieser Folien 1A und 1B abwechselnd und deckungsgleich übereinander, so entsteht ein Führungsbauteil 6, wie es in der Draufsicht schematisch aus Fig. 2 hervorgeht. An die beiden von den Schenkeln des Dreiecks gebildeten Flächen 6A, 6B sind die Zufuhrkammern 7A, 7B für die Fluide A', B' angeschlossen. An den mittleren Bereich 6C der gegenüberliegenden Fläche 8, in den die Kanäle 3A, 3B parallel zueinander ausgerichtet ausmünden, ist die Mischkammer 9C angeschlossen, über die die miteinander zu vermischenden oder zu dispergierenden Fluide A', B' bzw. deren Reaktionsprodukte C' abgeführt werden.

Die Schenkelflächen 6A, 6B sind gegenüber der Fläche 8 bzw. den beiden Seitenflächen 10, 11 des Führungsbauteils 6 so geneigt, daß die vom mittleren Bereich 6C der Fläche 8, an den die Mischkammer 9C angeschlossen ist, ausgehenden, von Folie 1A zu Folie 1B abwechselnd zu der Zufuhrkammer 7A und der Zufuhrkammer 7B führenden Scharen von bogenförmigen Kanälen 3A bzw. 3B annähernd gleiche Längen aufweisen.

Bei kreisbogenförmigen Nuten bzw. Kanälen bedeutet dies, daß die Bogenlänge

$$L = \frac{2\pi r \cdot a}{360^\circ} = \text{const.}$$

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sein soll, wobei r den jeweiligen Krümmungsradius und α den zugehörigen Bogenwinkel der betreffenden Nuten bzw. Kanäle bedeutet. Dies würde an sich zu gekrümmten Schenkelflächen 6A, 6B führen. In der Praxis ist es jedoch im allgemeinen ausreichend, diese gekrümmten Flächen durch eine Näherungsgerade "einzuebnen", z. B. nach der Vorschrift

$$\frac{\alpha_2}{\alpha_1} = \frac{r_1}{r_2}$$

wobei r_1 den größten und r_2 den kleinsten Radius und α_1 bzw. α_2 die zugehörigen Bogenwinkel zur äußersten und innersten Nut bedeuten.

Nach dem Übereinanderschichten werden die Folien 1A, 1B mit Deckplatten 15 versehen und vakuumdicht und druckfest miteinander verbunden, z. B. diffusionsverschweißt, so daß das so entstandene Führungsbauteil 6 die Gestalt eines homogenen Mikrostrukturkörpers erhält, der an die Kammern 7A, 7B und 9C angeschlossen wird.

Zur Herstellung der Folien 1A, 1B kann eine drehbare Sintermetallplatte 12 mit geschliffener Oberfläche eingesetzt werden, auf der eine Rohfolie 13 aus einem Metall wie z. B. Kupfer oder Silber mittels Unterdruck aufgespannt wird (siehe Fig. 3). In die sich drehende Rohfolie 13 werden mittels einem Formdiamanten (nicht dargestellt) in einem mittleren Bereich zwischen Drehmittelpunkt M und Peripherie der Rohfolie 13 eine Schar konzentrischer Nuten 14 eingearbeitet. Sodann wird die Rohfolie 13 in vier Einzelfolien 1A, 1B unterteilt in der Weise, daß vier mit dem mittleren Durchmesser der konzentrischen Nutenschar übereinstimmende Längsseiten 8, von deren mittlerem Bereich die kreisbogenförmige Nutenschar 14 ihren Ausgang nimmt, je zwei, also insgesamt acht Schmalseiten 10, 11 sowie je zwei Schenkelpaare 6A, 6B entstehen, wobei die Nutenschar 14 jeweils in einen der beiden Schenkel 6A bzw. 6B ausmündet. Es entstehen also aus vier Segmenten die vorerwähnten vier deckungsgleichen Fünfecke. Die Nuten haben vorzugs-

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weise rechteckige Querschnitte. Es können auch mehrere genutzte Rohfolien übereinander geschichtet und gemeinsam in die Fünfecke zerschnitten werden.

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Bezugszeichenliste

- 1A Folie
- 1B Folie
- 2A Nuten
- 2B Nuten
- 3A Kanäle
- 3B Kanäle
- 4A Zwischenstege
- 4B Zwischenstege
- 5A Nutböden
- 5B Nutböden
- 6 Führungsbauteil
- 6A hintere Stirnfläche
- 6B hintere Stirnfläche
- 6C mittlerer Bereich
- 7A Zufuhrkammer
- 7B Zufuhrkammer
- 8 vordere Stirnfläche
- 9C Mischkammer
- 10 Seitenflächen
- 11 Seitenflächen
- 12 Sintermetallplatte
- 13 Rohfolie
- 14 Nuten
- 15 Deckplatte
- A' Fluide
- B' Fluide
- C' Reaktionsprodukt
- r₁ Krümmungsradius
- r₂ Krümmungsradius
- α₁ Bogenwinkel
- α₂ Bogenwinkel
- M Drehmittelpunkt

Patentansprüche:

1. Statischer Mikrovermischer mit einer Mischkammer und einem vorgeschalteten Führungsbauteil für die getrennte Zufuhr von zu mischenden oder zu dispergierenden Fluiden zu der Mischkammer, wobei
das Führungsbauteil aus mehreren, plattenartigen, übereinandergeschichteten Elementen zusammengesetzt ist, die von je einer Reihe langgestreckter, benachbart zueinander verlaufender Kanäle durchzogen sind;
dabei sind wenigstens zwei Arten von Elementen A und B vorgesehen, die abwechselnd übereinandergeschichtet sind und deren Reihen von Kanälen mit ihren an die Mischkammer angrenzenden Mündungen übereinander liegen und dort einen gemeinsamen Querschnitt bilden, wobei abwechselnd die Reihen von Kanälen der Elementart A zu einer Zufuhrkammer eines Fluids A' und die Reihen von Kanälen der Elementart B zu einer Zufuhrkammer eines Fluids B' führen, gekennzeichnet durch folgende Merkmale:
 - a) die plattenartigen Elemente A und B für das Führungsbauteil bestehen jeweils aus dünnen Folien (1A, 1B) mit einer Dicke von 30 bis 1000 µm, vorzugsweise < 250 µm, bei einer lateralen Ausdehnung im Millimeterbereich, in die je eine Schar benachbarter Nuten (2A, 2B) eingearbeitet sind, so daß beim Übereinanderschichten der Folien (1A, 1B) je eine Reihe von Kanälen (3A, 3B) für die Führung der zu mischenden Fluide A' und B' entsteht;
 - b) die Nuten (2A, 2B) haben Tiefen von < 1000 µm, vorzugsweise < 250 µm, Breiten von 10 µm bis in den Millimeterbereich, vorzugsweise jedoch < 500 µm, bei Wanddicken der Zwischenstege (4A, 4B) und Nutböden (5A, 5B) von < 1000 µm, vorzugsweise < 250 µm;
 - c) die jeweiligen Scharen von Nuten (2A, 2B) in den übereinandergeschichteten Folien 1A, 1B verlaufen jeweils bogenförmig gekrümmt und abwechselnd von der Mischkammer (9C) zu je einer Zufuhrkammer (7A, 7B) für das Fluid A' bzw. B' in der Weise, daß alle Scharen von

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Nuten (2A, 2B) der Folien 1A, 1B parallel zueinander ausgerichtet in die Mischkammer (9C) ausmünden.

2. Statischer Mikrovermischer nach Anspruch 1, gekennzeichnet durch folgende Merkmale:
 - a) die übereinandergeschichteten Folien 1A, 1B haben in der Draufsicht die geometrische Form eines Fünfecks, das durch ein Rechteck mit hieran angeschlossenem gleichschenkeligem Dreieck gebildet wird, wobei eine Seite des Rechtecks die Grundseite des gleichschenkeligen Dreiecks bildet;
 - b) an die beiden von den Schenkeln des Dreiecks gebildeten Flächen (6A, 6B) sind die Zufuhrkammern (7A, 7B) für die Fluide A' bzw. B' und an die von der gegenüberliegenden Längsseite des Rechtecks gebildete Fläche (8) die Mischkammer (9C) angeschlossen,
 - c) die Schenkel (6A, 6B) des Dreiecks sind gegenüber seiner Grundseite so geneigt, daß die von der Mischkammer (9C) ausgehenden, von Folie (1A) zu Folie (1B) abwechselnd zu der Zufuhrkammer (7A) bzw. der Zufuhrkammer (7B) führenden bogenförmigen Nuten (2A, 2B) jeweils annähernd gleiche Längen aufweisen.
3. Statischer Mikrovermischer nach Anspruch 1, dadurch gekennzeichnet, daß die übereinandergeschichteten Folien (1A, 1B) zu einem vakuumdichten und druckfesten Mikrostrukturkörper (6) miteinander verbunden, z. B. diffusionsverschweißt, sind, der an die Zufuhrkammer (7A bzw. 7B) und die Mischkammer (9C) angeschlossen ist.
4. Verfahren zur Herstellung der Folien nach Anspruch 2, gekennzeichnet durch folgende Schritte:
 - a) eine Rohfolie (13) wird mittels Unterdruck auf eine drehbare, geschliffene Metallsinterplatte (12) gespannt;
 - b) in die sich drehende Rohfolie (13) werden mittels einem Formdiamanten in einem mittleren Bereich der Rohfolie (13) zwischen deren Drehmittelpunkt (M) und deren Peri-

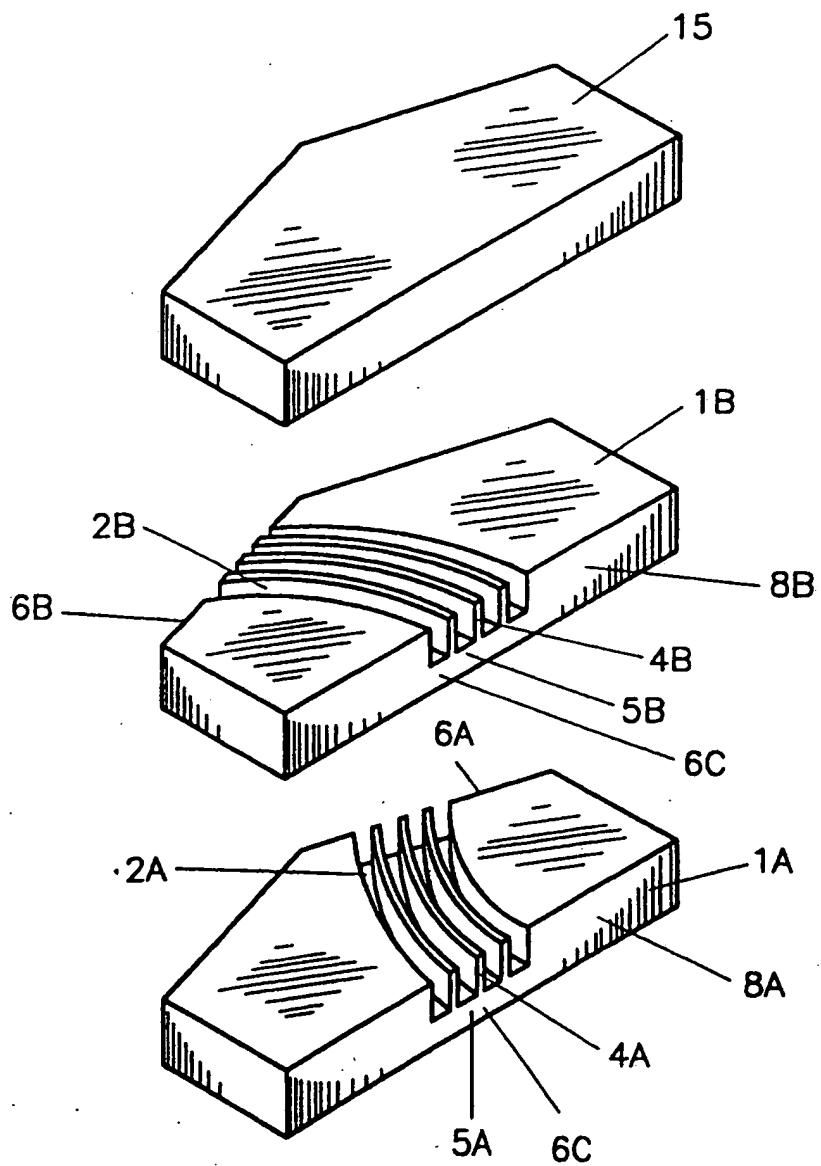
- 10 -

pherie eine Schar konzentrischer Nuten (14) eingearbeitet;

c) die Rohfolie (13) wird in vier Einzelfolien (1A, 1B) unterteilt in der Weise, daß durch zwei aufeinander senkrecht stehende, sich im Drehmittelpunkt (M) kreuzende Schnitte vier Segmente entstehen, und daß durch weitere Schnitte an jedem Segment vier deckungsgleiche Fünfecke gemäß Anspruch 2 gebildet werden.

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Fig. 1



2/2

Fig. 2

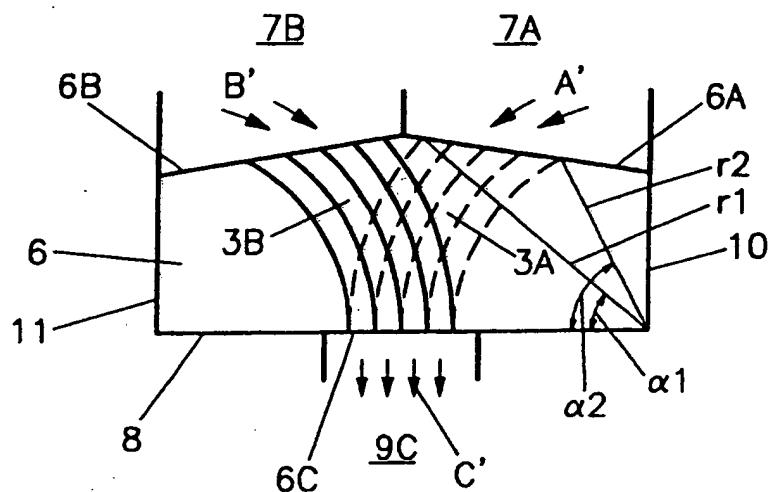
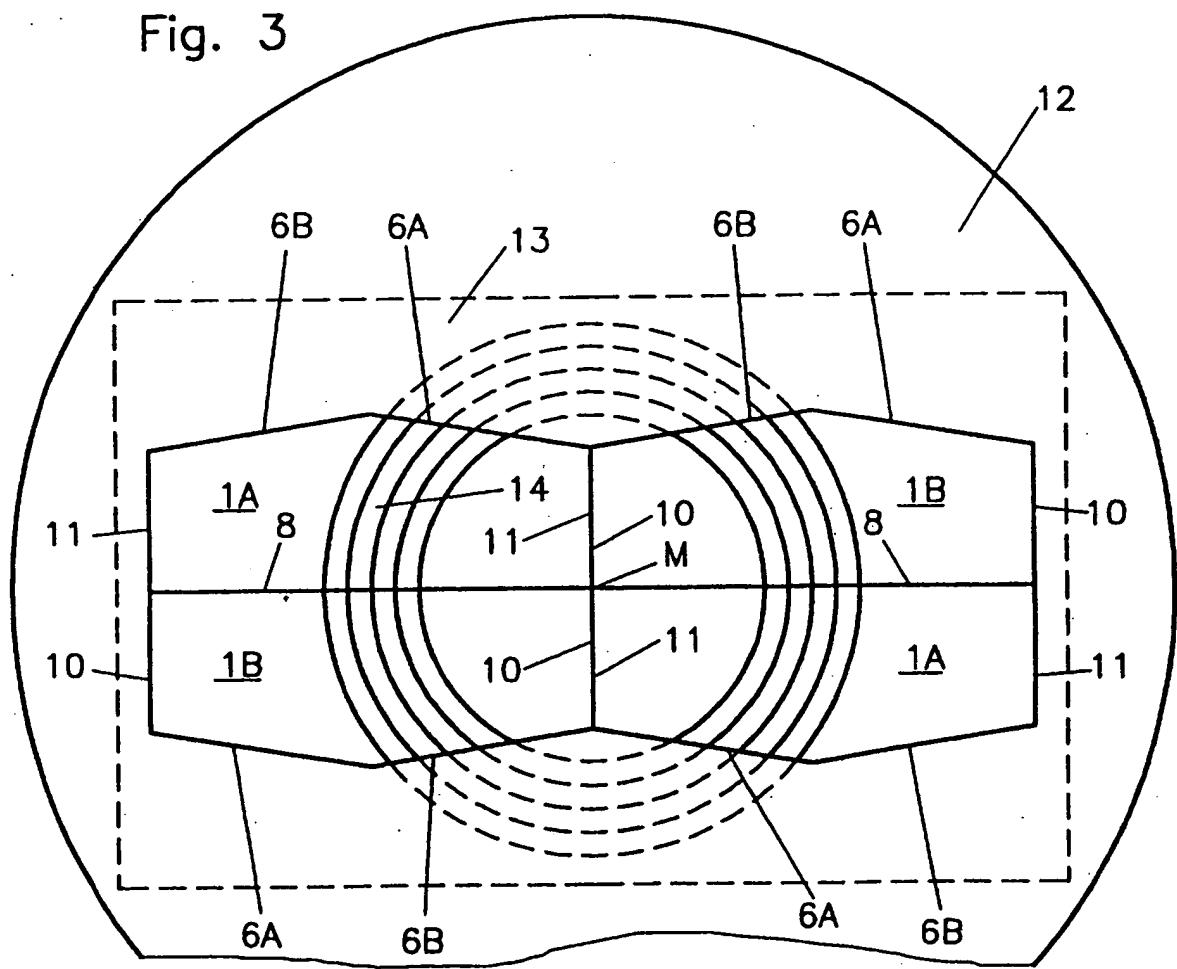


Fig. 3



INTERNATIONAL SEARCH REPORT

Int'l Application No.
PCT/EP 96/03162

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 B01F5/02 B01F5/06 B01J19/24

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
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C. DOCUMENTS CONSIDERED TO BE RELEVANT

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| A | PATENT ABSTRACTS OF JAPAN vol. 6, no. 149 (C-118), 10 August 1982 & JP,A,57 071627 (TEIJIN LTD), 4 May 1982, see abstract --- | 1 |
| A | WO,A,94 21372 (E.I.DU PONT DE NEMOURS AND COMPANY) 29 September 1994 see claims 1,21,28 --- | 1,4 |
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Date of the actual completion of the international search

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INTERNATIONAL SEARCH REPORT

Inv. no. and Application No.
PCT/EP 96/03162

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| Category | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
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INTERNATIONALER RECHERCHENBERICHT

Internationales Aktenzeichen
PCT/EP 96/03162

A. KLASIFIZIERUNG DES ANMELDUNGSGEGENSTANDES
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Recherchierte Mindestprustoff (Klassifikationssystem und Klassifikationssymbole)
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C. ALS WESENTLICH ANGESEHENE UNTERLAGEN

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Datum des Abschlusses der internationalen Recherche

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Name und Postanschrift der internationale Recherchenbehörde
Europäisches Patentamt, P.B. 5818 Patentamt 2
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Fax (+ 31-70) 340-3016

Bevollmächtigter Bediensteter

Cordero Alvarez, M

INTERNATIONALER RECHERCHENBERICHT

| | |
|-----------------|--------------------|
| Inte | nales Aktenzeichen |
| PCT/EP 96/03162 | |

C (Fortszung) ALS WESENTLICH ANGESEHENE UNTERLAGEN

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Int. nationales Aktenzeichen

PCT/EP 96/03162

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